Website for viewing photographs and video <a href="http://sas-origin.onstreammedia.com/origin/lubrizol/SANDBOX/110091PatentPgA/pages/IndexB.html">http://sas-origin.onstreammedia.com/origin/lubrizol/SANDBOX/110091PatentPgA/pages/IndexB.html</a>

## Forsberg:

- 1. The document Office Action of March 2010 is incorrect on a number of points.
  - a. The office action describes the additive invention as "magnesium containing liquid dispersion". The word "dispersion" is not mentioned once in the Forsberg patent. Forsberg correctly identifies what he has made as "highly basic magnesium complexes of sulfonate, carboxylates or phosphate". The terms "dispersion" and "complex" have clear definitions and are distinct. It is true that the Forsberg products are probably non-stoichiometric materials. Forsberg is making homogeneous phase solutions not dispersions, which by their nature are heterogeneous phases.
  - b. Component B is an oleophilic reagent and NOT a surfactant. There is a great difference. A surfactant acts at the interfaces relying of physical forces of attraction and repulsion to stabilise the colloidal system. A reagent chemically reacts and is change forming a new product.
  - c. Component B is limited to reactive reagents. The present invention favours surfactants which do not form a Mg complexes. If a Mg complex is accidentally formed it is only of value if it acts as a surfactant. It is the surfactant property which is important.
  - d. Water (Component C) is an important reagent in the Forsberg patent and he says so a ratio of 0.7:1 to 3:1 of water to Mg must be present throughout the process.
  - e. Ratios: Frosberg greatly emphasises the importance of the ratios of components:
    - i. (A): (B) [Mg base to Oleophilic reagent] of at least 5:1 (low) to 150:1 (high)
    - ii. (C) to (A) [Water to Mg base] of 0.7:1 (min for MgO) to 3:1, These ratios would seriously compromise the max solids that can be incorporated into the formulation. A 50% MgO dispersion would require 35% water (for 0.7:1 ratio); for the higher rati (3:1) 25% MgO would require 75% water leaving no room for any other materials.

The Frosberg recipe would because it is not a high solids system. The highest conc. solids is 26.91% (example 14) and that's after water and other volatiles have been removed.

- f. All the products made by Forsberg are gels not free flowing low viscosity fluids.
- g. Forsberg defines the reagents and detailed process changes in a multi-step process to achieve the desired product. The system he describes is 'open' he adds materials and removes material, by separation, distillation, filtration, and centrifuge. The present invention under discussion is a 'closed' system what goes in stays in (with one exception where optionally a dehydration step is included).
- h. The Office Action of March 2010 is factually incorrect. Last line of P3 to line 4 of P4. Describes an example:

233 parts Of Mg(OH)2 + 600 parts of alkylbenzenesulfonic acid are heated to 80oC for 2 hours wherein a gel is formed. 602 parts of this gel is diluted with 200 parts of toluene, centrifuges, and toluene stripped at 160-170oC. The correct process is (omitted parts are delineated):

233 parts of Mg(OH)2 + 600 parts of alkylbenzenesulfonic acid <u>and 1250</u> parts of water are mixed and heated to 80oC over 2 hours forming a gel.

The mixture is allowed to stand and 830 parts of aqueous layer removed.

570 parts of toluene is added and 300 parts of water removed by azeotropic distillation. 602 parts of the resulting gel are mixed with 200 parts of toluene, centrifuged, and then toluene is removed at 160oC-170oC <u>leaving</u> a gel.

These differences highlight the importance of water in the process and exemplifies that the products are gels.

# ·· Crawford et al.

This patent is relates to the preparation of specific fuel additives, Na, K salts, chiefly carboxylates, carbonates and borates. The materials are dispersions. Two routes at preparing the 'dispersions' are outlined:

- 1. The emulsion route. The water soluble metal salt, solubilised in water and then dispersed in an oil phase with the help of a surfactant (to stabilise the emulsion). The water is then removed (boiled off).
- 2. The text refers to a "reaction between an alkali metal carbonate-overbased carrier soluble alkali alkali metal sulphonate with boric acid to form an alkali metal borate reaction product". This attempts to use the overbasing technology to prepare the borate additive.
- a. I know this work well it does not work. The key approach was the emulsion route and they never succeeded in making a stable emulsion / dispersion. The traces of water cause large crystal to form (the traces of water were sufficient for Ostwald ripening to occur).
- b. The optimum surfactant for stabilising the emulsion is usually not able to stabilise the dispersion.



- c. This technology is limited to water soluble salts.
- d. The other well-known problem with the emulsion route is that the concentration of metal is very low you are limited by the saturation concentration in the water phase which is then stripped.

#### Rathon et al

This patent deals with making Mg(OH)<sub>2</sub> finely dispersed as a dry powder, a suspension or slurry for fire extinguisher applications. In fact they resort to the same milling technology we have used (Dyno mills). The differences are significant.

- 1. The dispersions are aqueous they cannot use combustible (organic materials for obvious reasons). Water is low boiling relative to base oils used in our invention and easy to remove.
- 2. The concentration of solids is low typically 20% mass.
- 3. The surfactants are water soluble materials e.g. sodium stearate.

Our technology is averse to water as their system is averse to organic materials. The whole principle of stabilisation is different - the HLB of our surfactant systems is much lower.

### Young (GB1061161)

This patent relates to the preparation of high concentration solids (40 to 85%), low viscosity (pumpable) using milling techniques to reduce the particle size to 2-10µm (even better (2-4µm). The surfactant is preferably a C1 to C20 carboxylate (whole range of carboxylates are covered). The extend the claims to other metal inorganic compounds (not Lithium). The focus is hot and cold corrosion applications with fuel oil combustion.

This is very similar to our patent - the differences are:

- Particle size primarily we are below 1µm.
- Surfactants we claim carboxylates our main work was with pibsa derived materials and sulphonates.
- > Surface area of the material we are working with is higher (not LiOH.H2O of course)

### Magyar (US 5851961)

This patent relates to an oil-in-water emulsions. The dispersed oil phase comprises an overbased metal containing carboxylate or sulfonate with surfactant and oil. The aqueous system contained a dispersant and an associative thickener. These are used as water based drilling muds.

The examiner picked up the preferred HLB of a polyacrylate surfactant as having an HLB of 10 to 19 (clearly water soluble). Therefore we deduced the surfactant range of 2 to 16 (in our claim 4).